METHODS OF TESTING FOR WATER RESISTANCE OF BITUMINOUS PAVING MIXTURES

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by

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Joint Highway Research Project

PURDUE UNIVERSITY
LAFAYETTE INDIANA

TECHNICAL PAPER

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BITUMINOUS PAVING MIXTURES

TO:	K. B. Woods, Director Joint Highway Research Project	April 10, 1958
FROM:	H. L. Michael, Assistant Director	File: 2-4 Project: C-36-6

Attached is a technical paper entitled, "Methods of Testing for Water Resistance of Bituminous Paving Mixtures." This paper has been prepared by Prof. W. H. Goetz of our staff. It is to be presented as part of a Symposium on the Effect of Water on Bituminous Paving Mixtures at the annual meeting of the American Society for Testing Materials in Boston, Massachusetts, in June, 1958.

The paper is presented for the record.

Respectfully submitted,

Harold L. Michael

Harold L. Michael, Assistant Director
Joint Highway Research Project

HLM:acc

Attachment

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Introduction

The problem of securing and maintaining adhesion between asphalts and aggregates in the presence of water has been recognized by asphalt technologists almost from the beginning of asphalt road construction. However, the development of test procedures and the use of them to study factors affecting the water resistance of bituminous paving mixtures dates from about 1930 or possibly a few years earlier.

Early investigators such as Ebberts (1)*, Davis and Curtis (2), Nicholson (3), Riedel and Weber (4), Dow (5), and Oberbach (6) developed and/or used simple laboratory test procedures in attempts to measure [illegible] water-oil preferential tests, wash tests, and boiling tests with and without electrolytes. At about the same time, and with considerable impetus later, physico-chemical concepts were applied in attempts to evaluate basic factors bearing on the problem.

Thus, the work of Nicholson (3), [illegible] (7), [illegible] (8), and [illegible] (9) can be cited. In their work the concept of surface activity or interfacial tension at solid-liquid interfaces as influenced by surface tension, wetting, adsorption, chemical reaction, polar molecules, and general surface energy relationships is discussed. In this connection, measurements were made of surface tension by bubble methods, wetting by measurements of contact angle, adhesion tension by capillary rise, and interfacial tension by volume determination of free-settling powders.

* Numbers in parentheses refer to bibliography

Although such basic measurements have been useful and necessary to an understanding of the problem and for the development of means to improve water resistance, they are not well suited to the everyday problem of determining whether or not a specific combination of bituminous material and aggregate will have satisfactory resistance to water in service, of determining the potential stripping resistance of aggregate or asphalt, or of determining the efficacy of treatments proposed for improvement of water resistance or resistance to stripping.

Since this early beginning, hardly a year has passed in which the technical literature does not report several studies covering many phases of the water-resistance problem. This interest continues to the present time, both with respect to the development and promotion of additives to improve coating and adhesion and with respect to the development of better test methods.

It is the purpose of this discussion to review these test methods and the technical literature concerning them in an attempt to classify and evaluate the test procedures with respect to their utility as a measure of water resistance. The test methods may be classified as follows.

1. Tests which measure surface activity or interfacial tension.

2. Coating or mixing tests in the presence of water.

3. Static immersion tests.

4. Immersion tests employing agitation --- wash tests.

5. Water displacement tests employing hot or boiling water with or without electrolytes.

6. Tests which measure change in volume on exposure to water --- swell tests.

7. Tests which measure strength or strength properties before and after water exposure.

8. Simulated traffic tests --- [illegible]

These may be grouped for the purposes of this discussion into simulated traffic tests, tests on compacted specimens, and tests on coated aggregate.

Simulated Traffic Tests

There is little doubt but that laboratory tests of the track or loaded-wheel type, in which there is some simulation of the road condition, have provided results of resistance to water action that correlate most closely with actual service. Such tests have several obvious advantages over other laboratory testing procedures. Those that have been or are being used vary from circular tracks large enough to carry a full-scale truck tire and wheel to a small-scale laboratory machine with wheels less than one foot in diameter and moving with a reciprocal motion.

Mack (10, 11) used a circular track test as early as 1938 to study the effect of water on bituminous mixtures. Samples were placed in the track, cured, and subjected to wheel traffic while being flooded with water. The number of revolutions or coverages of the wheel, at different temperatures, required for failure was taken as a measure of resistance to water. Holmes (12) and Klinger, Holmes and Phillips (13) in 1939 and 1940 also made use of a circular track test in a similar manner. It is interesting to note that these authors made use of the track test not only as a test method to evaluate stripping resistance and the effectiveness of addition agents to both aggregate and asphalt, but also as a controlled "field test" to provide data with which to compare the results of several small-scale laboratory tests. Likewise, Goldbeck (14) in 1949 used the results of a laboratory track test to evaluate the immersion-compression test and to recommend that the soaking period for the latter test be increased. The Road Research Institute of Sweden also has used a road machine to investigate the stripping problem and to evaluate anti-stripping agents (15).

These track tests have the advantage that bitumen-aggregate combinations may be tested in the specific mixtures in which they are to be employed, placed and cured in a manner simulating field construction. This includes application types of construction such as seal coats and surface treatments as well as mixture types. The testing of both hot and cold mixtures is possible, and all types of aggregate and bituminous material may be included. The testing action is similar to that of traffic, but conditions may be controlled as compared to an actual service test. On the other hand, these tests require large-scale special testing equipment, large amounts of material are involved, and the test procedures are complex and time-consuming. They are not well-suited to routine testing as required by many highway departments.

In recent years, the British have attempted to develop a small-scale bench-type of wheel or track test (16, 17, 18), apparently with considerable success. Specimens tested in this immersion wheel-tracking test are approximately 12 in. by 4 in. and about 1-in. thick formed by a laboratory rolling-compaction machine. The test wheels are 8-in. in diameter equipped with solid rubber tires 2-in. wide. The specimens are tested until failure occurs by moving the wheels (weighted) over them with a reciprocating motion at a rate of 25 times per minute during immersion in water at 40 C. A graphical record of penetration of the wheel into the specimen is made, and the time in hours required for collapse of the specimen is measured. This test apparently retains many of the advantages of the track-type of test while eliminating many disadvantages.

Tests on Compacted Specimens

In recognition of the importance of testing the whole aggregate at the bitumen content intended for use and in the paving mixture as a structural unit, and in recognition of the complexities of wheel-track tests, investigators of the water-resistance problem turned to the testing of mixtures in compacted specimen form. Such tests have included swell tests, tests for compressive, tensile or flextural strength before and after water immersion, cone penetration stability tests, abrasion tests on immersed specimens, and an "immersion tray test" for application to surface-treatment types.

As early as 1934, Stanton and Hveem of California (19) modified and standardized a so-called "swell test" developed by the Arizona Highway Department (20). In this test, a compacted specimen in a mold is exposed to water and the vertical swell measured. This method of test is in current use by these states. Also, the American Association of State Highway Officials has standardized such a method under AASHO Designation T101-42 (21). The method provides for determining the swell characteristics of dense-graded mixtures intended for use and for classifying dense-graded aggregates on a uniform basis with respect to their swell characteristics.

It is difficult to determine when strength tests on compacted mixture specimens were first used to evaluate water resistance, but Riis of Denmark referred to an adhesion test in which the compressive strength of a sample was determined after immersion in water in reporting to the International Road Congress in 1938 (22). Neumann (23) used compressive, tensile and flexural strength tests on samples stored in air and in water and compared wet and dry strengths as measures of water resistance in work reported in 1941. In this country, Krchma and Loomis (24), being interested in a quantitative method, reported in 1943 on water resistance studies in which loss in compressive strength by water immersion was used for the determination.

Present use of the compression test as a measure of water resistance was established by the work of the Bureau of Public Roads in which a large amount of research was done over a period of several years (25, 26, 27, 28, 29, 30). As a result, this form of testing has come to be known as the immersion-compression test and the method has been standardized by the American Society for Testing Materials as ASTM test method D1075-54, "Effect of Water on Cohesion of Compacted Bituminous Mixtures" (31). The test method has also been adopted by AASHO as test method T165-55 (32).

In addition to the tensile and flexural tests of Neumann (23) mentioned above, other strength or stability tests have been used in a manner similar to the compression test for the evaluation of water resistance by comparing test values on specimens before and after water immersion. Thus, Swanberg and Hindemann (33) used an abrasion test and Duriez and Arrambide (34) made use of a cone penetrometer stability test. Also, some highway agencies utilize their routine strength tests for bituminous mixtures in making measurements of water resistance by performing such tests on specimens before and after water exposure. As an example of this, the use by the State of California of cohesiometer and stabilometer values on specimens subjected to water vapor exposure (35) can be cited. In this connection, the immersion tray test developed by the Road Research Laboratory (London) (36) and used for the examination of adhesion agents for use in surface treatment work may be mentioned.

With respect to tests for water resistance in which compacted specimens are tested, one of the most recent studies is that of Andersland and Goetz (37) in which sonic testing techniques are applied to beam specimens to measure progressive loss in sonic modulus of elasticity caused by exposure to water. This method of test is shown by the authors to have considerable utility and to have inherent advantages over other types of tests since it employs specimens which contain materials of the same kind, gradation, and proportions compacted in a manner similar to that used in actual field construction. In addition, it possesses all of those benefits that accrue to methods employing a non-destructive type of test.

The use of tests which employ strength or strength properties to measure water resistance of compacted specimens has several advantages as compared to other methods. First, the test results are in terms of a quantitative measure, eliminating the need for visual ratings or estimates of performance. Also, the whole aggregate or mixture can be tested in a manner similar to that in which it will be used. On the other hand, such procedures are limited for the most part to dense-graded aggregates or mixtures, and, with reference to the standardized immersion-compression test in particular, to mixtures made with asphalt cements. In addition, these test methods are subject to all of the difficulties which arise in attempting to make mixtures and fabricate test specimens that are identical so that comparisons between wet and dry test values will be valid. Such test methods have not been correlated, particularly in a quantitative way, with field test results, although this may be due to the necessity for establishing realistic exposure conditions for the laboratory rather than to any basic deficiency in the test approach involved (14).

Tests on Coated Aggregate

While simulated traffic tests and tests employing compacted specimens have many advantages, they are complex and time consuming as compared to those tests in which the coated aggregate is subjected to water action in a loose condition. Therefore, this type of test is in considerable demand and is being widely used by highway testing agencies (38).

Tests of this kind were among the first to be utilized for the purpose of measuring the adhesion of bituminous materials to aggregate in the presence of water. Riedel and Weber (4) placed a sample of specified grain-size aggregate coated with bituminous material in boiling water for one minute and noted whether or not separation of the bituminous material from the aggregate occurred. In a modification of this method, Riedel and Weber substituted a sodium oleate solution for the water. In an attempt to make the test quantitative, they used increasing concentrations of soap and of electrolytes. Thus, coated aggregate is boiled in water for one minute. If the coating is not displaced, the test is repeated using sodium carbonate solutions of gradually increasing concentration until displacement occurs. The stronger the sodium carbonate solution required for displacement, the greater is the adhesion of the bituminous material for the aggregate.

In the boiling test, the use of boiling water has been justified on the basis that the viscosity of the bituminous material is thus reduced so that its importance is minimized. Criticism has been directed at this test method because in employing aggregate of fixed small grain size, aggregate must be broken in the usual case, thus creating freshly broken aggregate with characteristics different from those of the weathered material. Also, it is contended that there is no basis for employing electrolytes such as sodium carbonate in the measurement.

Nicholson (3) used a test in which the coated aggregate was immersed in water at 140 F and agitated for a specified time and the extent of uncoating determined. Dow (5) employed a similar test in which a sample of the loose mixture was placed in distilled water in a flask and shaken for periods of 1, 3, 5, 10, 15 and 30 minutes with examination for cloudy water and stripped sand grains after each shaking period. A static-immersion stripping test was developed in Germany (4) which was known as the German U37 Test in which the job grading of aggregate, or 3/4-in. aggregate in a modified test, were subjected to immersion in distilled water and a stripping value determined on a scale of 0 (completely uncoated) to 10 (completely coated).

These basic water exposure tests, commonly referred to as static-immersion or wash tests depending upon whether or not agitation is involved, have been widely used, alone and in combination, and have been modified in such use by many investigators of the water-resistance problem. In a 1937 committee report, Kelley (39) reported on several proposals for modifying the Nicholson test. Tyler (40) used a modified Dow wash test in his studies reported in 1938. Hubbard (41) has described an immersion test in which the severity of the test is increased by increasing the time and temperature of static immersion. Gzemski (42) has pointed out the importance of the pH of the water in any of these water immersion tests.

In 1937, Saville and Axon (43) reported on work in which they showed that results of the Nicholson test, a modified Nicholson test which included soaking, and the Riedel and Weber test did not correlate well with field test results. In the same year, Winterkorn, Eckert and Shipley (44) compared results of wash tests and boil tests with and without sodium carbonate and concluded that wash tests were more satisfactory. In further work, reported in 1939, Winterkorn (45) concluded that the best available methods for testing resistance of bitumen to stripping by water are the various modifications of the wash test in which quantitative data are obtained by varying conditions of the test. He pointed out the importance of standardizing the actual test procedure to insure definite equilibrium between the mineral surface and the gaseous or liquid phase with which it comes in contact. Podesta (46) compared the characteristics and utility of several immersion methods including Riedel and Weber, German DIN specifications, Highway Research Board, and the Nicholson wash test. In reporting on results of laboratory tests to evaluate anti-stripping admixtures, a report to the American Society for Testing Materials (47) concluded that the static-immersion test when used alone may give misleading results if the additive is not tested with the aggregate and asphalt with which it is to be used.

Although many of the immersion tests described have some utility in evaluating the water-resistance problem, they do not determine the actual adhesion to be expected with the binder and/or aggregate because the materials are not tested under conditions which will exist in the actual construction. Those methods which do not test the whole aggregate are open to criticism for this reason. Many test only the coarse aggregate, whereas when sand and filler are used they frequently play a dominant part in determining the water resistance of a mix. The tests do not measure the effect of water on the paving mixture as a unit. In these methods, the degree of stripping is evaluated visually and, therefore, may not determine that which actually exists.

Probably the most serious deficiency of such tests, particularly with respect to the use of them as indicator or comparative tests for aggregates, asphalts or additives for surface-treatment types of construction, relates to visual estimation of the stripping involved with the resultant variations due to the human element. Subcommittee B-26 of ASTM on "Effect of Water on Bituminous Coated Aggregates" has been working for a number of years in an attempt to standardize a visual estimation method (48). In this effort, the major concern has been for a method applicable to mixtures made with liquid bituminous materials. Their work covered three phases:

1. A survey and review of published methods to determine test conditions most generally used and to develop a test procedure based upon this review.

2. Cooperative tests to improve the test procedure and define conditions necessary for best reproducibility. A provisional method has been written (48).

3. A study of visual estimation methods and a survey of the limitations of the method.

From this work, it was concluded that the visual estimation method can be used provisionally at the 95% coated or better level, but precision below that level down to 5% coated is entirely unsatisfactory. Several methods were employed to improve the estimation method, including the use of standard-area photographs, weighted-average evaluation, and 100 percent coated evaluation, but without appreciable success.

As a result, Subcommittee B-26 and others have been active in trying to develop quantitative rating methods not subject to the human element. A dye absorption technique has been tried in which the bitumen-aggregate mixture is contacted with a standard solution of dye such as Safranine B (48). Since the uncoated aggregate will absorb the dye whereas coated aggregate will not, a basis is established for measuring uncoated area by measuring resulting dye concentration by colorimetric methods. The use of radioactive tracer techniques also has been employed (48, 49, 50). Radioactive calcium, usually in the form of calcium chloride, is applied to the aggregate surface before the aggregate is coated. The radiation intensity of the immersion water is a measure of the aggregate area accessible in the water exposure. Both the dye and radioactive tracer techniques usually give values indicating more stripping than is observed visually because aggregate area that is covered may not be adherent. Also, the radioactive tracer technique has been criticized because the aggregate surface is changed by application of the calcium chloride.

Enüstun (51) reported on a method of measuring the degree of stripping by measuring the amount of light reflected onto a photo-electric cell from the surface of the coated mixture. However, measurement of oblique reflectance has proved unsatisfactory; and vertical reflectance measuring equipment is being tried (48). Such methods suffer because the asphalt itself reflects light in various degrees depending upon the surface condition and aggregates vary in reflecting power because of aggregate color and texture variations.

Lortscher, Snyder and Filbert (52) have explored both indirect and direct methods of measuring stripped area. The indirect methods included measurement of dye absorption (fluorescein), leaching of some material from the aggregate, and dissolving of some material placed on the surface of the aggregate prior to coating. Direct measurement included visual estimation, optical methods using polarized light, and mechanical integration methods using a microscope and a motor-driven stage. Of the methods employed, the authors felt that the optical method using polarized light showed much promise and should be investigated further.

Brown, Sparks and Marsh (53) have suggested the use of a tracer-salt method in which lithium in the form of the chloride is used with determination of lithium in water in concentrations of 1-20 parts per million by the flame photometer. They present data to show that the method is objective, that diffusion of salt through an adhering asphalt film is negligible, that the concentration of stripping salt employed exerts no significant influence on the stripping of the system, and that the repeatability of results is very satisfactory.

Craig (54), in a recent study, has attempted to evaluate the variables in the static-immersion stripping test. He has concluded that, in spite of its shortcomings, the test has much to recommend it. It is simple, can be performed with a minimum of equipment, and is not too time-consuming. If the variables of the test are understood thoroughly and controlled to the degree necessary, it should serve as a valuable tool until such time as equally rapid test methods are devised that are more satisfactory. Craig states that, for the foreseeable future, it appears that the major test method for determining the stripping characteristics of bitumen-aggregate mixes will continue to be the static test or some variant thereof. The appendix to his paper presents a summary of the State Highway Departments anti-strip test requirements.

Summary

Over the past twenty-five or thirty years, there have been developed, both in this country and abroad, many methods of test designed to measure or evaluate the resistance of bituminous paving mixtures to the deleterious effects of water. This deleterious action, if present, commonly is revealed by a decrease in the bond (adhesion) between aggregate and bituminous material, by the development of uncoated aggregate area, or in extreme cases, by complete separation of bituminous material from the aggregate. The tests applied to this problem have varied from basic measurements of interfacial tension to simulated traffic tests.

Each of these test methods has features of merit, but some deficiencies as well. Deficiencies associated with arbitrary coating and curing conditions, artificial water exposure, exposure conditions for accelerated action, etc., are common to many of those considered. Many test methods do not test the whole aggregate, and many give only qualitative results that provide for relative measures of water resistance. Most of the test methods do not provide test results that correlate with actual field performance.

Nevertheless, coating tests, static-immersion tests, and wash tests have been utilized widely for evaluating the influence of such factors as aggregate type, asphalt type and grade, and the efficacy of asphalt additives. Although these methods, in general, employ only coarse aggregate predominantly of one size, a more serious deficiency is the dependence upon visual estimation for test results. Recognition of this has resulted in considerable effort to devise means of applying precision measurements. These include photographic techniques, measurement with photo-electric cells, radioactive tracer techniques, and the utilization of a water soluble salt in conjunction with a flame photometer. There is no standard ASTM test procedure covering the static-immersion type of test at this time, but a proposed method has been drafted which has now progressed to the voting stage.

Quantitative tests are those which measure change in volume or which measure strength or strength properties before and after water exposure. The best known of these is the so-called immersion-compression test which has been standardized by ASTM under designation D1075-54, "Effect of Water on Cohesion of Compacted Bituminous Mixtures." In addition to being a method producing quantitative results, this method has the further advantage that it applies to the whole aggregate and to the whole mixture that may be proposed for use. It is a method which requires the molding of a rather large number of specimens for any one determination, and it is not as simple a test as some demand. It applies only to mixtures containing penetration grade asphalts. Recently, a test method has been reported which utilizes sonic testing techniques in the evaluation of water resistance of compacted bituminous mixtures. It possesses all of those benefits that accrue to methods employing a non-destructive type of test. However, these quantitative test methods are deficient in that results obtained with them, for the most part, have not been correlated to road service.

Test methods for which good correlation with road service is claimed usually are some type of simulated traffic test--commonly referred to as a track test. However, these require special equipment not normally found in highway and material laboratories and have all of the deficiencies of tests requiring large quantities of materials and long periods of time to perform. There remains, then, the need for a small-scale laboratory test of a quantitative nature which can be correlated with actual field performance.

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